Analysis of Beryllium samples at AWE

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Background.

- Previous presentations have considered the management of Be at AWE, and the collection of IH related data.

- This presentation considers:
  - Types of sampling at AWE,
  - The range of techniques used to analyse Be samples, and
  - A feasibility study on a new analytical technique - Microwave Plasma – Atomic Emission Spectrometry.

Types of Sampling.

- There are a range of sampling methods for beryllium at AWE;
  - Personal Air Samplers
  - Stack samples
  - Smear samples
  - Static Air samples

  - Nose blows samples
  - Wound monitoring swabs

Preparation Methods.

- Different types of preparation for the various types of samples:
  - PAS, SAS and smear samples:
    - paper filters – leached in 0.1% w/v potassium hydrogen sulphate: 2%v/v nitric acid solution.
    - glass fibre filters – leached in 0.1% w/v potassium hydrogen sulphate: 2%v/v nitric acid solution and centrifuged before analysis.
  - Stack samples:
    - paper filters – leaching in nitric acid and perchloric acid.
    - glass fibre filters – leaching in nitric acid and centrifuged.

Preparation Methods

- Wound Monitoring samples:
  - cotton bud swab – digested in nitric acid, perchloric acid and 0.1% w/v potassium hydrogen sulphate: 2% v/v nitric acid solution.
- Nose blow samples:
  - tissue samples – nitric acid and 0.1 w/v potassium hydrogen sulphate: 2% v/v nitric acid.

Comparison of Techniques

Technique	Flame Atomic Absorption Spectrometry (F-AAS)	Graphite Furnace Atomic Absorption Spectrometry (GF-AAS)	Inductively Coupled Plasma – Optical Emission (ICP-OES)	Inductively Coupled Plasma – Mass Spectrometry (ICP-MS)
Flame	Combustion air / acetylene	Combustion Nitrous oxide / acetylene	Argon plasma	Argon plasma
Limit of Detection (LOD)	<1 µg/L	0.05 µg/L	0.2 µg/L	1 – 10 ng/L
Linear Range	10^3	10^2	10^5	10^5
Precision	Short term 0.1 – 1%	Short term 0.5 – 5%	Short term 0.3 – 2%	Short term 1 – 3%
			Over 8 hours 2 – 5%	Over 8 hours < 5%
Interferences	Chemical	Spectral	Spectral	Spectral and ionisation

NB: ICP-MS is available, but not routinely used for Be sample analysis.

Analytical techniques used.

- Personal Air Samplers – Flame AAS
- Static air samplers – Flame AAS
- Stack samples – graphite furnace AAS
- Smear samples – Flame AAS / ICP-OES

- Nose blows samples – Flame AAS
- Wound monitoring swabs – Flame AAS

Microwave Plasma – Atomic Emission Spectrometry – Conditions.

- Potential replacement technique for Flame AAS.
- Six samples submitted for feasibility study with Agilent Technologies.
- Instrument was set up using a primary wavelength of 234.861 nm.
- Glass cyclonic spray chamber used for sample introduction in order to minimise signal to noise ratio.
- A 30 second integration period with two replicate samples in order to obtain best detection limits.
- Calibration standards range – 0 – 100 µg/L (2% nitric acid matrix).

Microwave Plasma – Atomic Emission Spectrometry – Results.

- Recovery of spiked Be sample on filter paper:
  - at 25 µg/L – 101.88%
  - at 0.5 µg/L – 103%
- Limit of detection – 3x standard deviation of 10 blank samples (2% nitric acid matrix):
  - 0.087 µg/L
- Low level precision – 10 replicates of low standard
  - 4.31% rsd

Microwave Plasma – Atomic Emission Spectrometry – Advantages.

- Better sensitivity and detection limits from feasibility study.
- Can make a cost saving – use of site supplied nitrogen.
- Eliminating the use of flammable and oxidising gases
  - (nitrous oxide and acetylene).
- Easy to use software, low maintenance of equipment.
- Can do multi-elemental analysis
  - Feasibility study included Trade Waste samples (MP-AES Vs ICP-OES)

Summary.

- Full range of analytical techniques available for IH and regulatory sampling.
- Offsite labs used to undertake non-core business eg core sampling for decommissioning.
- Commissioning of a Microwave Plasma – Atomic Emission Spectrometer is underway.
- Intention to achieve UKAS accreditation for our Be analysis techniques.

Questions?